An In-depth Experimental Study of Wax Deposition in Pipelines

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*Abstract***—**Shale oils are highly paraffinic and, consequently, can create wax deposits that foul pipelines during transportation. Several factors must be considered when designing pipelines or treatment programs that prevent wax deposition: including chemical species in crude oils, flowrates, pipes diameters and temperature. This paper describes the wax deposition study carried out within the framework of YPF Tecnolgía S.A. (Y-TEC) flow assurance projects, as part of the process to achieve a better understanding on wax deposition issues. Laboratory experiments were performed on a medium size, 1 inch diameter, wax deposition loop of 15 meters long equipped with a solid detector system, online microscope to visualize crystals, temperature, and pressure sensors along the loop pipe. A baseline test was performed with diesel with no added paraffin or additive content. Tests were undertaken with different temperatures of circulating and cooling fluid at different flow conditions. Then, a solution formed with a paraffin incorporated to the diesel was considered. Tests varying flowrate and cooling rate were again run. Viscosity, density, WAT (Wax Appearance Temperature) with DSC (Differential Scanning Calorimetry), pour point and cold finger measurements were carried out to determine physical properties of the working fluids. The results obtained in the loop were analyzed through momentum balance and heat transfer models. To determine possible paraffin deposition scenarios temperature and pressure loop output signals were studied. They were compared with WAT static laboratory methods.

*Keywords***—**Paraffin deposition, wax, oil pipelines, experimental pipe loop.

I. INTRODUCTION.

ONVENTIONAL and unconventional crudes are a CONVENTIONAL and unconventional crudes are a mixture of several kind of hydrocarbons: paraffins, aromatics, naphthenes, resins and asphaltenes [1]. Paraffins are alkanes with a carbon number greater than 20 and high molecular weight [2]. Paraffins with chains containing up to 72 carbon atoms have been detected in some unconventional crudes [2]. At high-temperature and high-pressure reservoir conditions, the paraffin molecules are dissolved in the crude oil, so to start the deposition process it is necessary that the flow temperature reaches values below the WAT somewhere.

Solid paraffin deposits at the walls of crude oil production and transport pipes reduce circulation capacity and, in some cases, can completely obstruct the flow area (Fig. 1). The formation of paraffin deposits has become a serious problem for the oil industry, which in extreme situations requires interrupting production to replace and discard the clogged pipe section. One case that has been reported in the North Sea, in which obstructions were so severe and occurred so frequently, that the platform was abandoned at a cost of approximately \$100,000,000 [3]. For this reason, the development of remediation techniques has become a central topic in flow assurance research. Some of the remediation techniques currently used are inhibitor injection, pigging, thermal isolation, and exothermic chemical reaction.

Fig. 1 Pipeline obstruction at YPF´s "Loma La Lata Norte" location

YPF Tecnología SA (Y-TEC) is the R&D company belonging to YPF SA (51%) and CONICET (49%) where some research studies [4], [5], have been carried out to understand the paraffin deposition process and provide support to apply remediation techniques.

 One research line developed at Y-TEC is to investigate both CFD and 1D models to simulate the paraffin deposition process to estimate the paraffin deposition thickness increase with time in transport pipelines. A model to simulate the process of formation of paraffin deposits in ducts using computational fluid dynamics (CFD) techniques was developed by Y-TEC [4]. This model was originally proposed for simulation of laminar or turbulent flows; however, its validation was performed only

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for a laminar case of bibliography. Its validation for turbulent cases [5] did not show a very good agreement with the Y-TEC available data at that time for turbulent cases, and further calibration of the model was delayed until having an experimental facility available to validate the numerical results.

1D models are currently used at Y-TEC to simulate the paraffin deposition and provide some information about the paraffin thickness increase and paraffin aging process for both laminar and turbulent cases.

Y-TEC has a paraffin deposition test equipment (Flow Loop

Wax-Eval SS200) available in the Flow Assurance Pilot Plant which can be used to validate both the 1D and CFD models in laminar cases, but also to develop some experimental research (Fig. 2). This loop was not available for research until end of 2022, so the first experiments were carried out now to get the know how to operate it. This paper shows the first work and results obtained with this flow loop, in preparation for more intensive use to validate models and gain some insight to the paraffin deposition process.

Fig. 2 Flow Loop Wax-Eval SS200

II.PHYSICAL MECHANISMS OF PARAFFIN DEPOSITION

At high temperature and pressure reservoir conditions, the paraffins are dissolved in the crude oil. When leaving the reservoir, the oil flow decreases on temperature and pressure. The temperature drop is the main cause of the decrease in the solubility of paraffins in crude oil. The decrease in pressure also affects the solubility of paraffins because the lighter components of crude oil vaporize, but it is a second order effect when compared with temperature changes.

A critical temperature is observed on the solubility curve of a conventional crude. This is the temperature of appearance of solid paraffin in crude oil known as "Wax Appearance Temperature" (WAT) which is about 45 \degree C on the particular case showed in Fig. 3. It is both necessary that the temperature in the flow is below the WAT and that the temperature of the tube wall is below the temperature of the flow to start the deposition process. Most of the WAT measurements utilize the change in the physical properties of the oil during the formation of solid wax crystals. In all techniques for WAT detection, wax precipitation is first induced by a controlled cooling of the oil sample. At a temperature below but close to the WAT, the "first few" wax crystals form, and the resulting change in physical properties can be captured by the appropriate instruments; the WAT temperature can thus be determined [6].

There are four physical mechanisms that result on the paraffin transport to the wall, whether it is in dissolved form or forming crystals: molecular diffusion, shear scattering (convection and turbulent diffusion), Brownian diffusion, and gravity deposition. The contributions of the last two are negligible [7]. Molecular diffusion is responsible for the transport of dissolved paraffin from the bosom of the liquid to the wall and is the mechanism that has the greatest contribution in the formation of the deposit [1]. The paraffin that solidifies within the liquid can be transported by mechanisms of convection and turbulent diffusion. The importance of deposition by these mechanisms is still the subject of research [7].

The mechanism of molecular diffusion deposition is initiated when the temperature of the crude oil near the pipe wall is below the WAT, the paraffin molecules precipitate and adhere to the wall. The solubility curve of paraffins in crude oil relates temperature to the concentration of paraffins dissolved in crude oil. Therefore, the radial temperature gradient in the pipe causes a radial gradient of the concentration of dissolved paraffins and these paraffins diffuse from the heart of the crude oil, at a higher temperature, towards the wall. As they approach the wall, paraffin molecules separate from the solution and contribute to the growth of the deposit. In agreement experimental observations show that the thickness of the deposit presents axial symmetry as the flow itself [1], [8], [9].

When deposited, solid paraffin particles enclose a portion of the liquid crude oil [1], [10], [11], turning the paraffin deposit into a porous medium whose pores contain liquid hydrocarbon. Due to the molecular diffusion mechanism, paraffins migrate from the flow to the interface between the paraffin deposit and the crude oil. Depending on the temperature at the interface, dissolved paraffins may precipitate at the interface or may diffuse and precipitate inside the paraffin deposit. This process is known as deposit aging and is characterized by an increase in the mass fraction of solid paraffin in the reservoir over time. Therefore, the aging mechanism produces a deposit of greater hardness and more resistant to remediation techniques.

There are few experimental studies that contemplate the formation of paraffin deposits in multiphase flows, either crudegas or crude-water. Sarica and Panacharoensawad [12] highlighted that the phenomenon depends on the flow pattern. This multiphase flow condition is above the scope of this work.

III. LOOP DESCRIPTION

Y-TEC acquired a flow loop to develop experimental studies of paraffines deposition process in pipes. This loop, called WAX EVAL SS200 flow loop, was built supplied and mounted in Y-TEC facilities by the French firm Vinci Technologies. Its main components are: (1) Pressurized tank, (2) Gear Pump, (3) Flowmeter and densimeter, (4) Heating system for hot pipes, (5) Pressure and temperature sensors, (6) Control and safety valves, (7) Refrigeration equipment (Chiller), (8) Solids Detector System (SDS), (9) High pressure microscope and (10) Bottle of inert gas (N2).

The circuit is designed to work at a maximum pressure of 150 bar and to withstand pressures of up to 210 bar. A schematic illustration of it is presented in Fig. 4. The temperature can reach up to 90 °C at the heated pipes. The components dimensioning and sealing must ensure these working conditions.

As seen from Fig. 4, the crude circulates from the container pushed by a gear pump, passing through a flowmeter and densimeter to enter the working section, node (i), where it exchanges heat with the coolant to the required conditions of the paraffin deposition process. Pressure and temperature are controlled throughout the circuit and at the output section, node (s), before re-entering the heated part.

The system has a countercurrent loop of a refrigerant fluid to control the crude cooling process (showed in dashed line in Fig. 4). A 50% solution of ethylene glycol is circulated through an annular pipe of 4" diameter, at a fixed flow rate of 60 L/min. The ethylene glycol temperature can vary from a threshold starting at -20 °C. A "Chiller" device transfers heat from this fluid to another refrigerant having a compressor unit outside the test room. The refrigeration system has a working power of 12 kW.

Fig. 4 Experimental loop scheme

To get the required pressure and temperature information, the test sections are instrumented with differential pressure gauges (between head points), absolute pressure gauges (at two points of the section in WAX EVAL 200) and platinum thermoresistance temperature sensors (at the head and intermediate points). A frequency meter in the pump makes it possible to determine the rotational speed of the impeller. The signals from these sensors are acquired by a digital acquisition board that acts as an interface with the computer software, which in turn records the data. Another set of sensors is used to perform the studies under preset conditions. The same plate allows a centralized control of the flow operation of the different fluids. The pressure to which the assembly is subjected is controlled manually from an inert gas cylinder with its respective regulator.

The measurement of the thickness of paraffins deposited in the loop are made from three alternatives:

A.Pressure Difference Measurement

This approach relies on studying the influence of internal diameter change over pressure drop. It is evident that an effective diameter changes due to paraffin deposition thickness δ_w will determine important variations of pressure drop.

Naming D_i the initial pipe diameter, the thickness can be obtained from the following expression:

 $T_{R,{\rm input}}$ $T_{R,\mathrm{output}}$ – annular center line

Fig. 5 Heat exchange scheme

$$
\delta_w = \tfrac{D_i}{2}\left[1-\left(\tfrac{\Delta p_f}{\Delta p_{f0}}\right)^{-4/19}\right]
$$

where Δp_f is the pressure drop after deposition and Δp_{f0} is the pressure drop before deposition. In (1), the exponent is obtained considering turbulent flow condition. This will require to be evaluated depending on the flow regime.

Considering errors on pressure measurement the sensitivity ϵ of this paraffin deposition thickness estimation is:

$$
\epsilon \frac{d\delta_w}{d\epsilon} = \frac{2\,D_i}{19} \Big(1+\frac{\Delta p_f}{\epsilon}\Big)^{-23/19} \left(\frac{\epsilon}{\Delta p_{f0}}\right)^{-4/19}
$$

Thus, the possibility of determining changes in diameter by this method is highly dependent of the quality of the pressure measurement and the signal/noise ratio. Particularly, among other aspects, the precision of the sensors used is critic. This calculation does not consider the effects of entering the cooling zone. Given the length of the duct (greater than 600 diameters), this effect is not considered as an important source of error in the calculations.

The paraffin deposition thickness that yields (1), is an average value along the length of the duct. Considering that a localized obstruction could lead to misleading results, it is better to carry out the calculation using the differential pressure sensor that records differences between input and output. Then it is required to check with the pressure difference values in intermediate parts of the loop. Pressure gradients must remain constant if the deposition is uniform.

The measurement of pressures with absolute sensors at intermediate points of the test section should however be carefully analyzed. Given the position of these sensors, their measurement may be contaminated by deposition of paraffin on the sensor and/or due to its intrusive nature as its extremity is in the current of crude oil.

B.Temperature Difference Measurement

The crude oil circulation loop together with the cooling system constitutes a countercurrent heat exchanger. The measurement of temperatures inlet and outlet of crude oil and refrigerant allow an alternative way to set the deposition thickness value. The principle on which they stand is that the deposition of paraffins adds an additional conductive resistance to the heat transfer process, which is proportional to the thickness of the wall and which, at the same time, modifies the internal radius for the calculation of the internal convection coefficient.

As the wax thickness increases, the deposit reduces the heat transfer between sample and cooling fluid. This added thermal resistance is approximately in direct proportion with the wax thickness layer.

The heat transfer equation is solved with measurement of

- Sample temperature drop over the pipe
- Pipe wall temperature.

The thickness of the deposit arises as a difference between the internal radii and the effective internal radii.

In general, this method is less accurate than the previous one but provides additional information.

C.Pump Revolutions Per Minute (RPM) Measurement

This method provides additional information. It leans on the fact that, by changing the passage section, the power of the pump required to drive the same flow will increase. The power of the pump (product of pressure difference, flow rate and efficiency) is linked with the speed of rotation of the pump from the characteristic curves of the bomb. Then, an increase in the pressure difference presents as a correlate an increase in pump rpm. Similarly, an increase in viscosity due to the formation of paraffin crystals involves an increase in required power and pump rpm. So, this measure is an indicator of the beginning of the deposition process*.*

IV. RHEOLOGY AND FLUID PHYSICAL PROPERTIES

Two fluid samples have been used in all the experiments:

- A Diesel sample: taken from YPF´s refinery in La Plata before additives are added to get a Diesel Fuel.
- A Diesel + Paraffin sample: prepared in the loop's tank. 2.6 kg of 55 °C fusion point wax was added to 45 liters of Diesel. Tank temperature was kept on 70 °C while stirring for 6 hours to ensure that paraffin was completely dissolved in the Diesel.

Fluid's density and Diesel viscosity were determined at different temperatures by Stabinger Viscometer according to ASTM D7042.

WAT was determined by two methods: DSC (Dynamic Scanning Calorimetry) and Viscometry.

DSC is a thermal technique that captures the heat of crystallization released by the sample upon wax precipitation. The analysis was performed using TA Instrument Discovery. All data acquisition and analysis has been achieved using the TRIOS thermal analysis software. Samples to be analyzed were heated to 70 °C in closed containers and shaken thoroughly to ensure complete dissolution of precipitated solids. They were then transferred to aluminum capsules and weighted. The samples were analyzed using cooling temperature programming. Temperature was as ramped to 75 °C and then cooled at a rate of -3 °C/min to -60 °C. Fig. 6 shows the thermogram obtained. The wax used has a marked WAT at 55 °C, blue curve. On the other hand, the Diesel has a smaller heat peak release at -11 °C, red curve. At this low temperature first diesel molecules start to crystalize. Finally, the Diesel +

Paraffin sample has a WAT of 23 °C. It is remarkable how the paraffin crystallization's temperature goes down from 55 to 23 °C when it is dissolved in the Diesel, confirming that all the components in the oil contribute to wax deposition. DCS results were also used to best estimate wax content in Diesel + Paraffin sample trough (3):

$$
\%Wax = \frac{Q_{Released}}{\Delta H_{crystallization}} 100\%
$$
 (3)

The heat released is get as the area under $Diesel + Paraffin$ sample and the Heat of crystallization was obtained as the area under the Wax thermogram. Wax content is 6%.

Fig. 6 DSC thermograms on Wax, Diesel, and Diesel + Wax samples at a cooling rate of 3 °C/min

Above the WAT, the crude oil behaves as a Newtonian fluid, and the viscosity of oil as a function of temperature can be described using an Arrhenius-type equation. As temperature is lowered below the WAT, the wax precipitates, and the precipitated solids remain suspended in the bulk liquid. The suspended wax particles change the flow properties of the crude oil. Determination of the WAT based on viscometry utilizes the change in the slope in the viscosity vs. temperature curve. It should be noted that when the temperature is further lowered below the WAT, a substantial amount of wax can precipitate and remain suspended in the crude oil, causing a non-Newtonian fluid behavior. The rheology analyses were performed using a doble gap geometry on TA Instruments Rheometer. The samples were poured on the geometry and heated to 60 °C. This temperature was kept for 30 min. Then temperature was ramped down at 12,5 C/h to 5 C while maintaining the shear rate. Results are shown in Fig. 7, where a sharp increase in viscosity and a transition from Newtonian to non-Newtonian is observed at 26 °C.

Finally, WAX deposition was also determined by the cold finger method. In this test, hot oil is exposed to a cold surface and after the fixed period the weight of wax deposited is determined. The equipment used for carrying out the test was a Techbox System. 200 ml of sample was poured in the deposition cell, the water bath was kept at 50 °C while metal fingers were at 0 °C during 24 hours. After that time, 2,24 g of wax were deposited when a Diesel + Wax sample was studied.

Fig. 7 Diesel + Wax viscosity as function of temperature at 300, 100 and 1 s^{-1} shear rates

V.FLOW LOOP TESTS

A.Pressure Diesel at Constant Flow, Variable Temperature

With a reference fluid, diesel of known properties, density and viscosity (ρ, μ) illustrated in Fig. 8 we study the ability of sensors to evaluate pressure drop for different temperature values. Tests were performed at constant flow rate G.

For the fixed flow value $G = 7.3$ L/min, we sought to calibrate the differential sensor signal with respect to the pressure drop along the Loop, at different temperatures like those indicated in Fig. 9. This sensor is the DP303 from Validyne Engineering, which is a true bidirectional "Wet-Wet" differential pressure transducer designed for applications where very low differential pressures are to be measured in fluid systems involving high line, or working, pressures up to 5000 psig.

Three periods of approximately constant temperature were maintained to have reference values in the calculation of physical properties.

Thermal sensors that correspond to sockets of the differential pressure sensor stand out among the multiple thermal sensors in the circuit (Fig. 10). Those are the TE-01 signal at the input of diesel to the Loop and the signal TE − 05 at the exit.

Temperature values at the inlet are always higher than the values at the output since it is the hot current of a heat exchanger. However, observing the curves of Fig. 9, during some transients the output signal has a higher temperature than that of the input. Qualitatively, the response in these transients seems more damped (slower times) at the input than at the output and could justify the difference.

The pressure drop between points 01 and 05 is estimated from the flow rate, diameter and physical properties of the fluid evaluated at the corresponding trial temperature. The time intervals used for the determination of average temperature values are indicated with symbols on the graph (Fig. 9). At the

end of the test, a temperature descent ramp is applied for the same flow rate, as seen from hour 3.

Fig. 8 Data and adjustment of physical properties of Diesel: (a) Density; (b) Viscosity

Fig. 9 Temperature and pressure differential sensor recording; the symbols indicate time intervals where the temperature remained constant; the grey line represents the voltage drop recorded by the DP303 sensor, whose filtered value is that of the black solid line

Fig. 11 shows the estimated values for the differential pressure from the calculation of the friction factor *f* in the pipe (in kPa) compared to the sensor signal in (mVolt). We determine *f* using the Clamond correlation [13]. The linear adjustment relationship between the pressure difference and the voltage output is $\Delta P_{15}/kPa$] = 54.1V − 9.92 as illustrated in Fig. 11 (a). On the other hand, Fig. 11 (b) indicates that the values of the Reynolds number obtained with this fluid are higher than the transition threshold for internal flow of Newtonian fluids Re > 2300. It should be noted that the normal working condition of the Loop with crude oil corresponds to flows with Reynolds numbers smaller than 2300, corresponding to the laminar regime.

B.Diesel WAT

The next test consisted of testing the ability of the Loop to determine the crystallization of diesel paraffins.

Fig. 10 Temperature sensors

Petroleum-derived diesel is composed of approximately 75% saturated hydrocarbons, mainly paraffins including isoparaffins and cycloparaffins, and 25% aromatic hydrocarbons, including naphthalenes and alkabenzenes.

Through the DSC test, the temperature value associated with the crystallization process is between -5 and -10 °C. On the other hand, the cloudy point of the temperature values for hot (diesel) reservoir streams is -7 °C (ASTM D 7698). Fig. 12 shows the evolution in time of the hot current Diesel (a) and the cold one (refrigerant, ethylene glycol 50%) (b) at the inlet and outlet of the Loop. Comparing both graphs, it is verified that the temperature of the refrigerant changes less since its thermal capacity, the product of the specific heat and the flow rate, is greater than that of the hot fluid.

During cooling, in Fig. 12 (a), a change in slope can be seen that produces an approach of the hot current curve to the cold current curve between 4 and 5 hours. The filtered differential pressure signal is presented together with the previous temperature signal in Fig. 13 (a).

Fig. 11 (a) Sensor pressures and values. (b) Runoff regimes

Fig. 12 Temperature values both at inlet and outlet for: (a) Diesel, (b) Coolant

Fig. 13 (a) Temperature and differential pressure signal; (b) Heat flux

Fig. 14 Reynolds number for test B

It can be seen on the sensor filtered curve that for times over 4.5 hours, there is an increase in the differential pressure drop. In addition, we can estimate the heat flow in each fluid stream according to:

$$
\dot{Q}_c = \rho_c \ G \ C_p \ (TE_{01} - TE_{06})
$$

Minimizing out-of-circuit losses, the heat flow of the cold current is $\dot{Q}_f \approx \dot{Q}_c$. The result is observed in Fig. 13 (b). First, for $t \approx 4h$ an increase in heat flow is observed, together with a change on flow regime. Then, for times higher than t_d , the flow decreases, which corresponds to an increase in the current thermal resistance. To analyze this, it is convenient to verify the type of flow that takes place in the duct. Then, based on the

density (*ρ*) and viscosity (*ν*) (properties corresponding to the average diesel temperatures for each time interval) we estimate the Reynolds number, $Re = U D/v$, presented in Fig. 14. Since the Reynolds number values observed are close to the critical value of change from laminar to turbulent regime in pipes, $2300 < R_e < 4000$, it is reasonable to consider changes in thermal resistance linked to hydrodynamic transition phenomena.

On the other hand, no crystals were observed under the

microscope, a measure that could better characterize the process. For these reasons, we therefore conclude that crystal deposition did not occur in the duct walls.

C.Diesel + Paraffin

2.6 kg of paraffin was added to the diesel bringing the mixture in the container to 70 °C to ensure a good dissolution. The physical properties of the mixture were surveyed and are shown in Fig. 15.

Fig. 15 Physical properties of Diesel+Paraffin solution: (a) Density; (b) Viscosity

Fig. 16 WAT of Diesel+Paraffin determination, test #1: (a) Temperature and pressure signal; (b) Heat flux

On a first test (test #1) a 10 C/h descent ramp was applied to reach up to 40 °C within the Loop. A first difference with the previous case is that the initial condition corresponds to the fluid at the temperature of 70 °C without refrigerant operation. Cooling begins and therefore the hot current temperatures at the inlet and outlet to the Loop begin to differentiate as shown in Fig. 16 (a). The differential sensor shows a moderate increase that corresponds to the variation of density and viscosity properties of the fluid. On the other hand, the heat flux monotonously increases without appreciating discontinuities of its behavior. The process follows the cooling ramp, but the time is not enough to distinguish a regime change.

A second test (test #2) was carried out repeating the previous conditions but allowing more cooling time. The corresponding curves are presented in Fig. 17. As it was tested for a longer time, a qualitative change in flow is observed.

A rise in the pressure difference can be distinguished from 6

hours by comparing the value with lower times. Greater precision can be observed in the change in temperatures between the inlet and outlet on Fig. 17 (a) which translates into an increase in effective heat flow as clearly seen in Fig. 17 (b). The two regimes are adjusted with lines, highlighting the difference in slope at time $t_d = 6h$. The temperature at the inlet corresponding to $t_d = 6h$ is 36.8 °C.

The corresponding time to time Reynolds numbers are presented in Fig. 18. The range is 4500 *< Re < 10000*, which corresponds to turbulent regime. Despite being more viscous that the Diesel previously tested, the mixture test #2 is carried out at higher temperatures and correspond to a lesser viscosity.

Although according to the Reynolds range the flow regime is turbulent, it is still possible that transition phenomena take place and modify the heat flux shown in Fig. 17 (b). Thus, for the flow rate adopted and the total time considered, it cannot be assured that the deposition phenomenon has been achieved.

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Fig. 17 WAT of Diesel+Paraffin determination, test #2: (a) Temperature and pressure signal; (b) Heat flux

Fig. 18 Reynolds number for test #2

VI. CONCLUSIONS

Despite that in the three proposed tests, the condition of wall deposition was not clearly reached, the test #2 with a mixture of Diesel and Paraffin shows changes in properties compatible with crystal formation. A measurable deposition could have been achieved only if the studies had been carried out for a much longer period. Testing with lower flow rates than those applied in this work would have promoted deposition, but the sensitivity of the instruments used imposes a limit to it.

So, further investigations need to be made allowing even more cooling time. Another important aspect is the fluid used for tests. There are some well-known synthetic crudes in literature that could be better suitable to run tests to detect paraffin disposition like the one used in reference [1] a synthetic crude mixture made up by a mineral oil (Blandol) and Kerosene with a carbon number distribution between C23 and C38.

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